

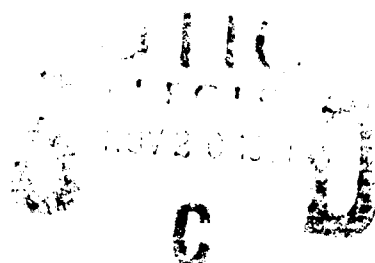
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US ARMY
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MTL TR 91-37

METAL INJECTION MOLDING OF TUNGSTEN HEAVY ALLOYS: SBIR PHASE I

October 1991

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FINAL REPORT

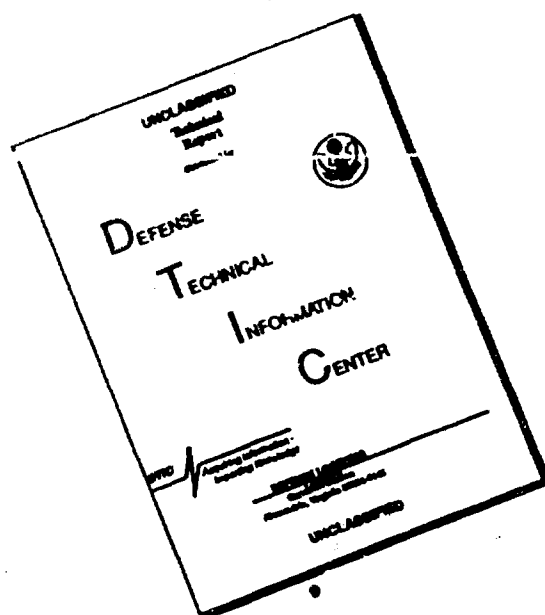
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ABSTRACT

The objective of SBIR A90-133 is to investigate the feasibility of injection molding tungsten heavy alloys into net or near-net shape parts. The focus of Phase I was to demonstrate (on a laboratory scale) that powder injection molding (PIM) can provide parts with equivalent or superior sintered material properties to those that have been achieved for press/sintering heavy alloys of similar compositions. In addition, geometric shrinkage(s) and key process variables were identified and analyzed.

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SBIR A90-133 FINAL REPORT

Metal Injection Molding of Tungsten Heavy Alloys

OBJECTIVE

The objective of **SBIR A90-133** is to investigate the feasibility of injection molding tungsten heavy alloys into net or near-net shape parts. The focus of **Phase I** was to demonstrate (on a laboratory scale) that **powder injection molding (PIM)** can provide parts with equivalent or superior sintered material properties to those that have been achieved for press/sintering heavy alloys of similar compositions. In addition, geometric shrinkage(s) and key process variables were identified and analyzed.

EXPERIMENTAL PROCEDURE

A sample test geometric shape was identified and a (injection molding) tool was constructed to prototype green parts. **Two feedstock formulations** with different volume fraction solid loadings were specified as test parameters. Test specimens were then debound, sintered, heat treated and tested for resultant material properties and alloy chemistry. This investigation was divided into **twelve tasks** that are identified in the following listing.

Task 1: Elemental Particle Analysis

All elemental particles were analyzed using **laser diffraction** analysis sizing hardware/software incorporated in a Malvern Master Sizer. They were also examined using an optical microscope.

Task 2: Powder Blending

Elemental W, Ni and Fe powders were **mechanically blended** in a V-blender to form a master alloy.

Task 3: Solid Loading Curve

The solid loading curve was constructed after adding small powder increments to the feedstock mixer which established the loading limits (dictated by the equipment) between 0.4 and 0.5. These were established as the two test sample loadings used in this investigation.

Task 4: Feedstock Rheology

Feedstock viscosity characteristics were established by the rheological profiles for both sample feedstock lots.

Task 5: Feedstock Mixing

Prototype feedstock was mixed in a Scott Turbon Mixer utilizing the master powder alloy and a three part thermoplastic/lubricant binder formulation.

Task 6: Prototype Test Mold

The prototype test specimen mold was produced and dimensionally modified by Diversified Mold, Inc. (Huntington Beach, CA).

Task 7: Injection Molding

Injection Molding was performed in a Arburg (Model 305-211-700) with test specimens provided for both solid loading volume fractions.

Task 8: Debinding (Chemical)

The green test specimens were chemically debound with 1-1-1 trichloroethane and thermally dried in a Bowden Liquid Turbo Charged system.

Task 9: Furnacing (Thermal Debinding and Sintering)

The parts were thermally debound, sintered and heat treated using furnacing cycles that sequentially interfaced atmospheres of dry and wet hydrogen, and vacuum.

Task 10: Part Dimensioning

The sintered parts were measured for width, length and thickness dimensions; and the shrinkage percentages were calculated.

Task 11: Material Property Testing

The resultant material properties for each volume fraction solid loading were established by obtaining the following parameters for each sintered sample set:

Density
Hardness
Microhardness
Tensile Properties
Matrix Volume Fractions
Grain Size

Task 12: Chemical Analysis

The alloy chemistry of the blended powder and the sintered samples were compared for **carbon content**. The **alloy chemistry** of both sintered sample lots was evaluated by Energy Dispersive Spectroscopic (EDS) analysis.

CONCLUSION

PIM is a **viable process** for producing **tungsten heavy alloys** since the resultant properties were comparable to press/sintered alloys having similar compositions. The **processing conditions** within PIM did not produce any significant changes in sintered density, hardness, microhardness, and the matrix alloy chemistry between samples. The **shrinkages** were different for the two volume fractions of solid loading, but the variation in the shrinkages for each set was within 1%. Chemical debinding when combined with the thermal debinding resulted in total binder removal in all samples without any **residual carbon**. The sintered densities, mechanical properties, and the microstructures were all similar to normal press/sintered alloys.

In conclusion, **Phase I** demonstrated that **PIM** can achieve production of near net shaped tungsten heavy alloys parts that result in little or no machining. Recent developments in alloying conventional tungsten heavy alloys suggest the feasibility of fabricating high strength, high hardness, near net shaped parts for numerous applications. The success of **Phase I**, provides a **strong base** for **further research** to develop military and commercial products based on tungsten heavy alloys.

RESULTS AND DISCUSSION

Elemental Powder Analysis

Technology Associates Corporation ("TAC") ordered elemental powders from the following sources:

W Powder	GTE Products Corporation Towanda, PA 18848
Fe Powder	GAF Chemical Corporation Linden, NJ 07036
Ni Powder	Nova Met Specialty Products Corporation Wyckoff, NJ 07481

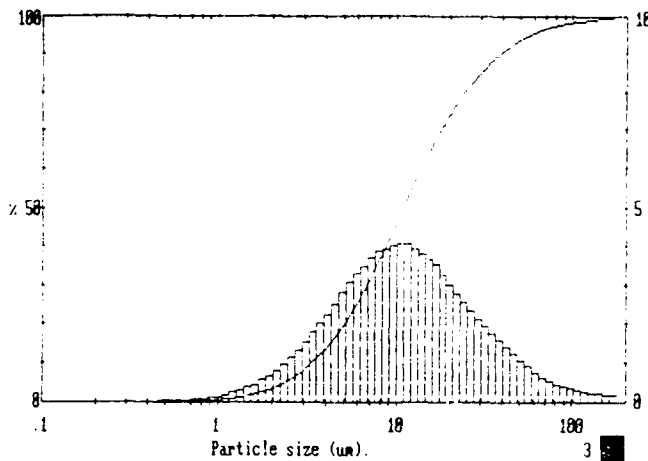
They were processed to determine particle size distributions. This was accomplished with a Malvern Master Sizer.

The results are summarized graphically and numerically in Figure 1. The following table lists the summary statistics taken from the histograms and cumulative distributions:

<u>Powder</u>	<u>Mean (μm)</u>	<u>Std Dev. (μm)</u>
W	10.82	9.50
Ni	10.28*	8.79
Fe	6.52	7.32
Alloy	12.43	9.99

* Takes into account a small portion of agglomerated iron particles due to the limited range of ultrasonic interface as illustrated in Figure 1 (lowest graph).

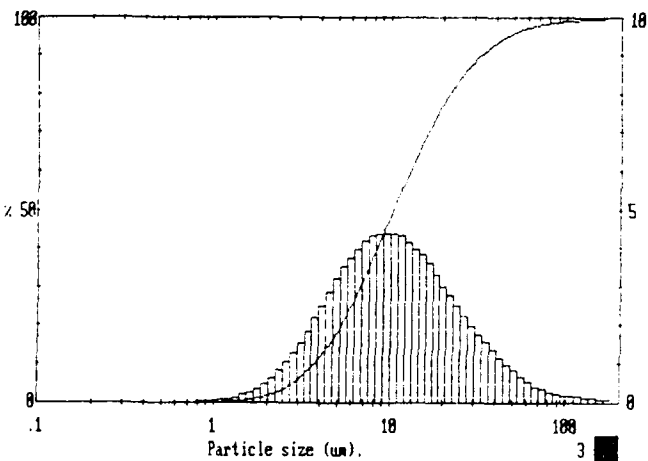
Figure 2 illustrates the interactive nature of the Fe powder in forming the master alloy.



SAMPLE DETAILS: TUNGSTEN

High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	Span
180	0.1	164.4	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
164	0.1	158.7	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
149	0.1	153.4	0.1	136	0.1	124	0.1	113	0.1	103	0.1	0.1
136	0.1	148.7	0.1	124	0.1	113	0.1	103	0.1	93.6	0.1	0.1
124	0.1	144.3	0.1	113	0.1	103	0.1	93.6	0.1	85.2	0.1	0.1
113	0.1	140.4	0.1	103	0.1	93.6	0.1	85.2	0.1	77.6	0.1	0.1
103	0.1	136.8	0.1	93.6	0.1	85.2	0.1	77.6	0.1	70.7	0.1	0.1
93.6	0.1	133.5	0.1	85.2	0.1	77.6	0.1	70.7	0.1			
85.2	0.1	130.5	0.1	77.6	0.1	70.7	0.1					
77.6	0.1	127.8	0.1	70.7	0.1							
70.7	0.1	125.3	0.1									

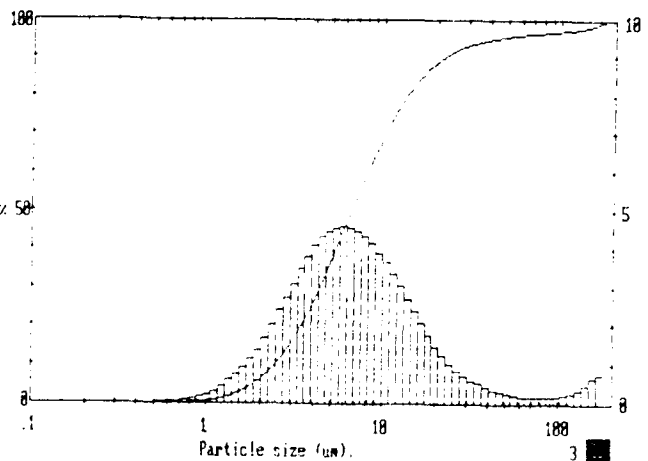
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 Volume distribution Sp.S.R 0.8522 #/cc.



SAMPLE DETAILS: NICKEL

High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	Span
180	0.1	164.4	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
164	0.1	158.7	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
149	0.1	153.4	0.1	136	0.1	124	0.1	113	0.1	103	0.1	0.1
136	0.1	148.7	0.1	124	0.1	113	0.1	103	0.1	93.6	0.1	0.1
124	0.1	144.3	0.1	113	0.1	103	0.1	93.6	0.1	85.2	0.1	0.1
113	0.1	140.4	0.1	103	0.1	93.6	0.1	85.2	0.1	77.6	0.1	0.1
103	0.1	136.8	0.1	93.6	0.1	85.2	0.1	77.6	0.1	70.7	0.1	0.1
93.6	0.1	133.5	0.1	85.2	0.1	77.6	0.1	70.7	0.1			
85.2	0.1	130.5	0.1	77.6	0.1	70.7	0.1					
77.6	0.1	127.8	0.1	70.7	0.1							
70.7	0.1	125.3	0.1									

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 Volume distribution Sp.S.R 0.7931 #/cc.

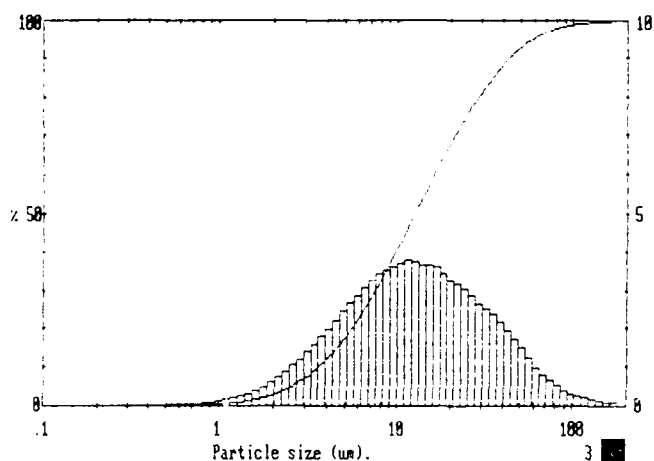


SAMPLE DETAILS: 140N

High Size	Under %	High Size	Under %	High Size	Under %	High Size	Under %	High Size	Under %	High Size	Under %	Span
180	0.1	164.4	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
164	0.1	158.7	0.1	149	0.1	136	0.1	124	0.1	113	0.1	0.1
149	0.1	153.4	0.1	136	0.1	124	0.1	113	0.1	103	0.1	0.1
136	0.1	148.7	0.1	124	0.1	113	0.1	103	0.1	93.6	0.1	0.1
124	0.1	144.3	0.1	113	0.1	103	0.1	93.6	0.1	85.2	0.1	0.1
113	0.1	140.4	0.1	103	0.1	93.6	0.1	85.2	0.1	77.6	0.1	0.1
103	0.1	136.8	0.1	93.6	0.1	85.2	0.1	77.6	0.1	70.7	0.1	0.1
93.6	0.1	133.5	0.1	85.2	0.1	77.6	0.1	70.7	0.1			
85.2	0.1	130.5	0.1	77.6	0.1	70.7	0.1					
77.6	0.1	127.8	0.1	70.7	0.1							
70.7	0.1	125.3	0.1									

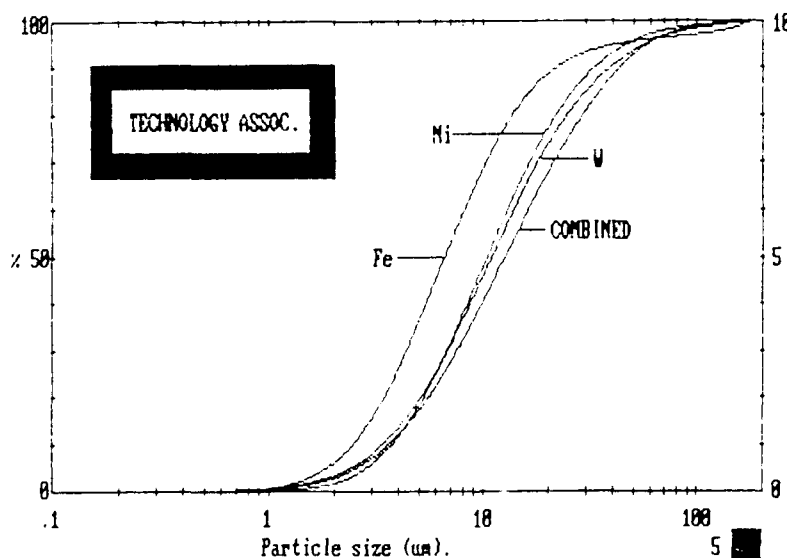
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 Focal length = 100 mm Residual = 0.16 %
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 Volume distribution Sp.S.R 1.1124 #/cc.

Figure 1. Malvern Elemental Powder Particle Size Distributions.



SAMPLE DETAILS: IRON-NICKEL-TUNGSTEN COMBINED

High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	High Size	In %	Span
180	0.1	164.4	1.0	123.0	3.2	18.25	1.4	2.95	1.3	1.46	0.1	0.14
164	0.1	58.7	1.2	21.0	3.3	17.51	3.3	2.69	1.1	10.96	0.1	19.17
149	0.1	53.4	1.5	19.1	3.5	16.84	3.1	2.45	0.9	10.88	0.1	
136	0.2	48.7	1.7	17.4	3.6	16.22	2.9	2.23	0.7	10.80	0.1	0.12
124	0.2	44.3	1.9	15.9	3.7	15.67	2.7	2.03	0.6	10.73	0.1	7.75
113	0.3	40.4	2.2	14.4	3.7	15.17	2.5	1.85	0.5	10.66	0.0	
103	0.3	36.8	2.5	13.2	3.7	14.71	2.2	1.69	0.4	10.60	0.0	
93.6	0.4	32.5	2.8	12.0	3.8	14.28	2.0	1.53	0.3	10.55	0.0	
85.2	0.5	30.5	3.0	10.9	3.7	13.91	1.8	1.40	0.3	10.50	0.0	
77.6	0.7	27.8	3.3	9.94	3.6	13.56	1.6	1.2	0.2			
70.7	0.8	25.3	3.1	9.05	3.5	13.24	1.4	1.1	0.2			
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Focal length = 100 mm Residual = 0.506 %												Div, 0.5
Presentation = 001% Observation = 0.0069 Volume Conc. = 0.0222												Div, 0.5
Volume distribution Sp.S.A. 0.7746 m ² /cc												Div, 0.5



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SAMPLE DETAILS: IRON-NICKEL-TUNGSTEN COMBINED

Figure 2. Malvern Alloy Particle Size Distribution.

Powder Blending

The elemental W, Ni, and Fe were **processed** in a roller mill and then **blended** for five hours in a V-blender to the following ratios:

<u>Metal</u>	<u>Weight (gm)</u>	<u>Weight Percentage(%)</u>	<u>Density(g/cc)</u>
W	19,000	95.0	19.30
Ni	800	4.0	8.90
Fe	200	1.0	7.87

The powders were studied using an optical microscope which displayed the great affinity of Fe particles for any other elemental powder upon contact. This resulted in powder agglomerates in the final alloy which was confirmed by the cumulative powder size distribution shown in Figure 2.

Solid Loading Curve

The metal powder master alloy was mixed with the TAC polymer binder in a **double planetary** Scott Turbon mixer, which had an oil jacket for heating the mixer basket. The solid composition was adjusted to provide a final heavy alloy composition of **95W-4Ni-1Fe** by weight. The binder was first liquified in the double planetary mixer and the elemental powder mixture was added gradually to the molten binder. The initial amount of solid addition was adjusted to yield a 0.3 volume fraction of solid loading. The mixing was continued at a temperature of 177° C (350°F) for 45 minutes after which small samples were taken out from three different areas of the molten feedstock. The density of these samples were carefully measured by water immersion technique. The solid loading was increased in small steps to afford different volume fractions in the feedstock. After each increment, the material was mixed before repeating the sample extraction and density measurement. A 0.5 volume fraction of solid loading was selected as a limit as result of mixer mechanical limitations.

The volume fraction of solid loading was calculated from the amounts of binder and solid used in the mixing process. The measured densities for all volume fractions are somewhat lower than the calculated densities. This could be expected as the mixing was accomplished in air. The viscous liquid mass often entraps air within its body and forms minute air bubbles. As the feedstock is cooled, viscosity of the mixture increases very rapidly and the air bubbles are permanently entrapped in the feedstock mixture. Longer mixing times or maintaining a **vacuum** could alleviate this problem. Utilizing a roughing pump vacuum during the mixing step is perhaps the best solution to this problem as it can then bias the diffusivity of the air away from the feedstock mixture.

One of the feedstocks contained **0.5 volume fraction** of solid. Since the viscosity of this mixture was felt to be quite high, it was decided to use a second feedstock with lower volume fraction of solid adjusted to 0.4. The point where the solid-binder mixture was no longer a viscous fluid and mixing became difficult was at 0.5. This was considered to be the **Critical Volume Fraction (CVF)** of solid loading. For subsequent investigations both feedstock loadings were tested.

Feedstock Rheology

The change in feedstock viscosity with increasing volume fraction of solid is a leading indicator of the CSL since viscosity relates the feedstock shear stress to the shear strain rate. A PIM feedstock that exhibits a visco-elastic nature and a high viscosity will make the molding process difficult. A capillary rheometer provides an excellent method for characterizing the PIM feedstock and was completed on the Rheometrics RDA2. There were two feedstock samples:

Heavy Loading (H.L.): 0.5 volume fraction solid.

Light Loading (L.L.): 0.4 volume fraction solid.

Both feedstock samples were broken, then ground, and finally compressed into 25 mm disks using disposable, serrated and parallel plates.

Both samples were tested in strain sweeps at 10 radians per second at 177°C (350°F) from 0.1% to 100% strain. The samples were also tested in frequency sweeps at 50% strain at 177°C (350°F) from 0.1 to 100 radians/second.

The results of these strain sweeps are plotted in Figures 3 and 4. The notation for each curve can be summarized as follows:

Symbol - Notation	Meaning	Units
G' (\diamond) - Upper Curve	Heat Dissipation per % Strain	DYN/cm ²
G'' (Δ) - Middle Curve	Elasticity per % Strain	DYN/cm ²
η^* (\sim) - Lower Curve	Viscosity per % Strain	Poise

The H.L. sample showed greater elasticity and structure throughout the test than the L.L. sample. Neither sample showed a linear visco-elastic region down to 0.1% strain. The H.L. sample became more viscous than elastic at 15% strain (Figure 3) while the L.L. sample at 8% strain (Figure 4). The H.L. sample also showed a higher viscosity and viscous modulus throughout the sweep. Finally, the H.L. sample was more strain dependent than the other sample; and thus, it may have been less stable.

The frequency sweeps were done at high strains to simulate processing. The H.L. sample showed higher viscosity at low frequencies for good melt strength, and lower viscosity at high frequencies for easier processing (more shear thinning). The L.L. sample showed less particle associations at higher frequencies. Both samples showed strong associated particles at very low strains. From the data it appeared that the H.L. sample might be easier to process but exhibit a less stable dispersion.

sbir heavy strain sweep 177c

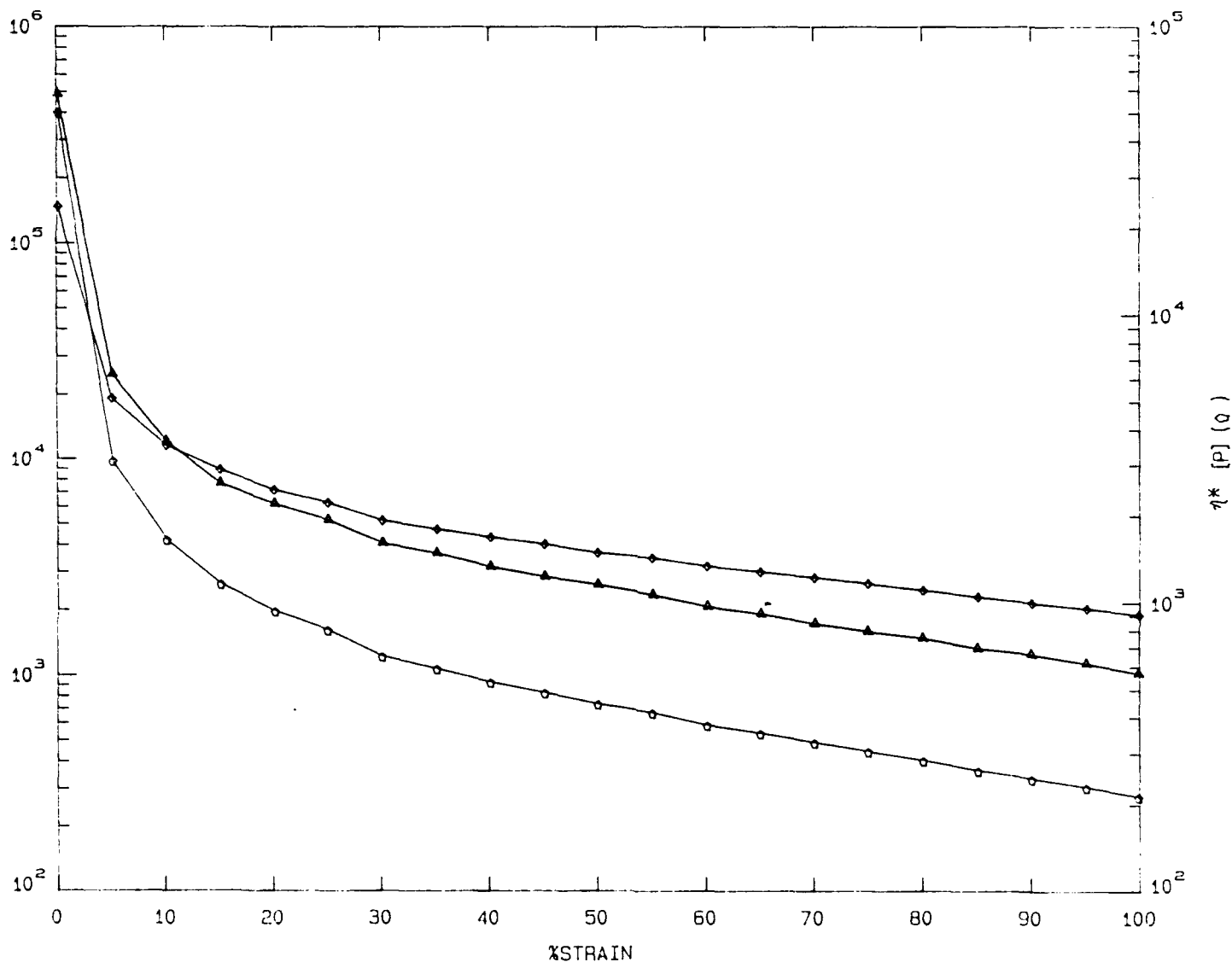


Figure 3. H.L. Feedstock Rheological Stress/Strain Curves at 350° F (177° C).

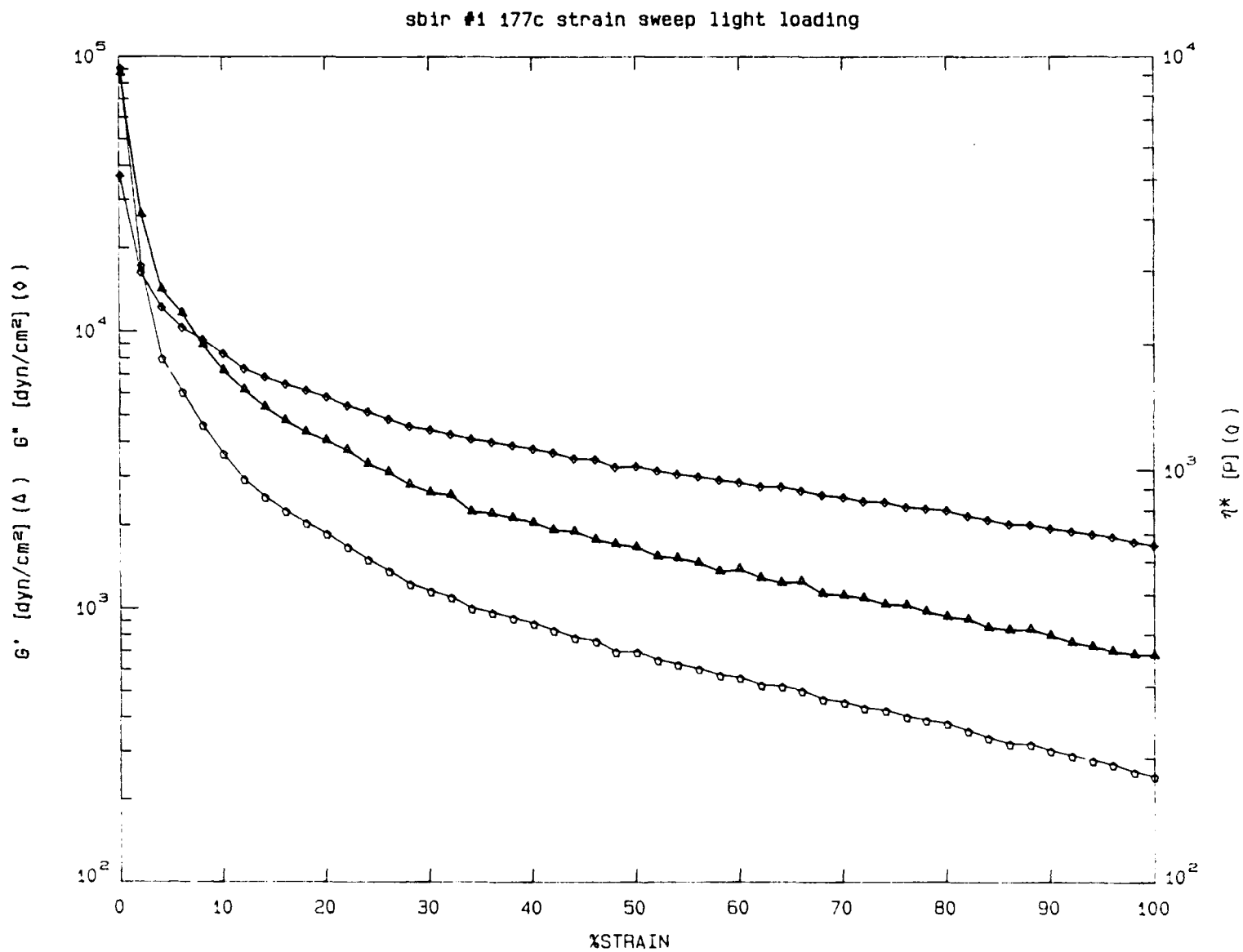


Figure 4. L.L. Feedstock Rheological Stress/Strain Curves at 350° F (177° C).

Feedstock Mixing

The master powder alloy and the binder matrix were mixed in an oil jacketed, Scott Turbon Double Planetary Mixer. The feedstock formulation was as follows:

<u>0.50 Volume Fraction Solid</u>	<u>Wt. (gm)</u>
W-Ni-Fe (18.18 gm/cc)	5,000.0
Polyethylene (HDPE) (.905 gm/cc)	67.2
Polyalphamethyl Styrene (1.075 gm/cc)	100.8
Lubricant (.913 gm/cc)	<u>168.0</u>
TOTAL	<u>5,336.0</u>

<u>0.40 Volume Fraction Solid</u>	<u>Wt. (gm)</u>
W-Ni-Fe (18.18 gm/cc)	4,440.0
Polyethylene (HDPE) (.905 gm/cc)	112.0
Polyalphamethyl Styrene (1.075 gm/cc)	168.0
Lubricant (.913 gm/cc)	<u>280.0</u>
TOTAL	<u>5,000.0</u>

Each binder ingredient was combined in air atmosphere at 177°C (350°F) and 15 psi (.103 MPa). The binder mixing was completed in 45 minutes, powder loading in 15 minutes, and the feedstock mixing in 45 minutes.

The feedstock was gradually cooled to room temperature and hand granulated.

Prototype Test Mold

The injection mold for the prototyped test specimen was produced by Diversified Mold, Inc. (Huntington Beach, CA). Figure 5 shows a schematic illustration and a dimensional diagram with the "green" test bar dimensions (in metric units) before sintering. The shape of the die has some **degree of complexity** as the gauge section gradually narrows down from the wider areas, where the specimen is usually gripped during tensile testing. This shape demonstrated the capability of the **feedstocks** to be injection molded and provide a test specimen for determining the tensile properties.

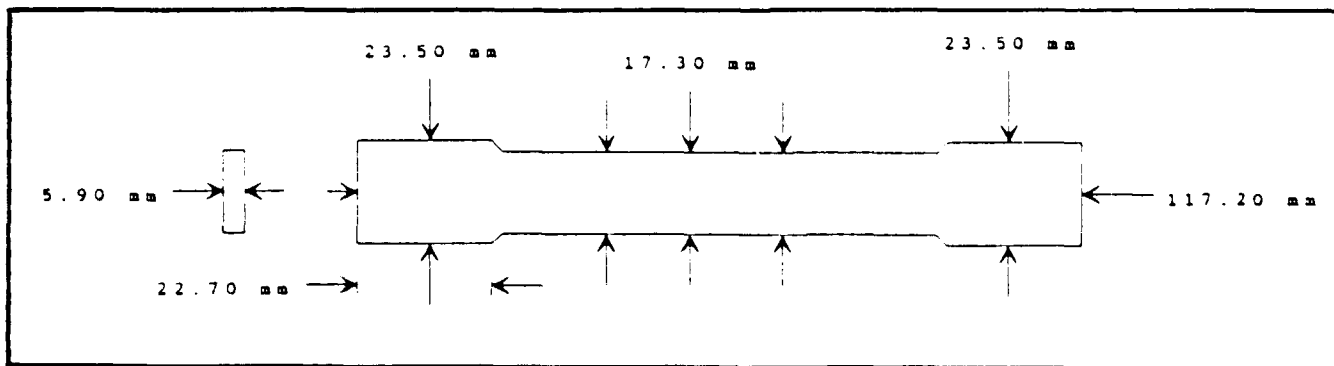
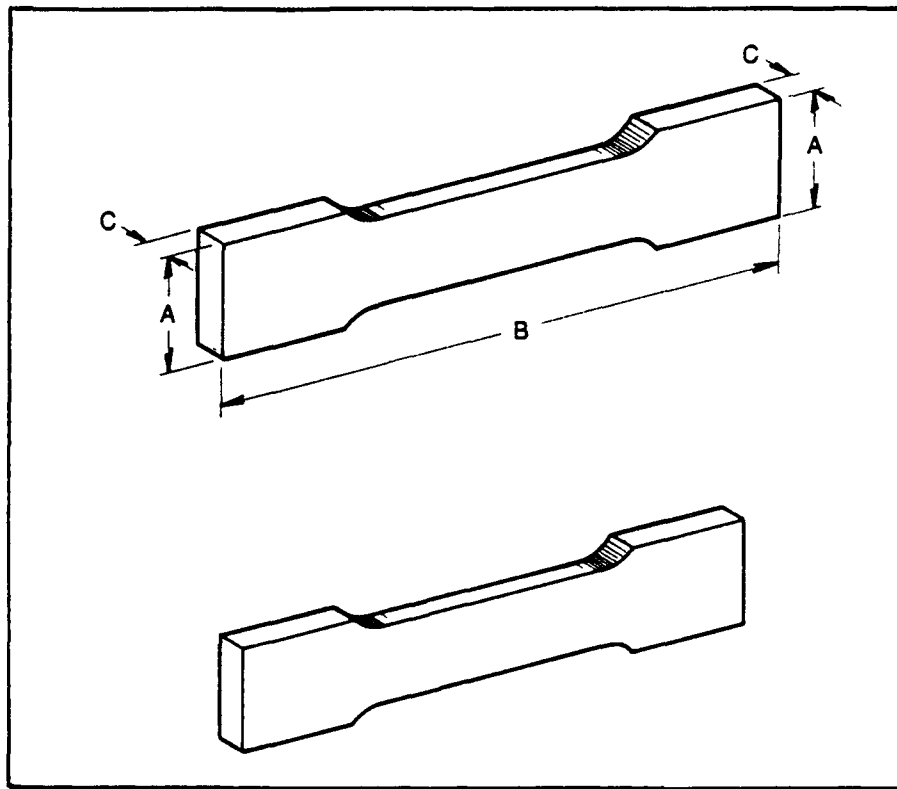


Figure 5. Illustration of Tensile Test Bar Specimen.

Injection Molding

The prototype test specimens were created in a Arburg Injection Molding machine (Model 305-211-700). The molding conditions were:

Temperature:	Nozzle - 177°C (350°F)
	Front - 177°C (350°F)
	Middle - 177°C (350°F)
	Rear - 171°C (340°F)
Time Profile:	Injection - 6 sec.
	Hold - 2 sec.
	Cool - 18 sec.
Pressure:	Injection - 500 psi (3.445 MPa)
	Hold - 500 psi (3.445 MPa)

A total of 22 test bar specimens were molded. Nine were 0.50 volume loading and thirteen were 0.40 volume loading. The injection molded green bars were carefully weighed and their dimensions measured along the sections shown in Figure 5, marked "A" (width), "B" (length), and "C" (thickness). Two widths, one length, and two thicknesses were measured for each green tensile bar to determine the uniformity of shrinkage after sintering. Inappropriate injection molding conditions can create the possibility of obtaining flow induced non-uniformity, which would be displayed in the shrinkage results.

Debinding (Solvent)

A combination of solvent and thermal debinding was utilized for two reasons: 1) Thermal binder extraction is very slow and can create excess gas pressure within green parts resulting in severe cracking and distortion, and 2) Thermal debinding can leave binder residuals which induce carbon contest in the sintered microstructure.

The TAC process sequentially extracts portions of the binders leaving enough to provide adequate debound part strength. The test bars were chemically debound for eight hours in 1-1-1 trichloroethane and dried for twelve hours using a hot air circulator in a Bowden Liquid Turbo Charged system.

Furnacing (Thermal Debinding and Sintering)

Eighteen (18) solvent debound test specimens were sent to GTE Products Corporation (Towanda, PA) and were sintered under the supervision of Dr. James Mullendore. **Four sets** of test samples were obtained:

- 1) L.L. Test Bars sintered at 1480°C (2,696°F).
- 2) L.L. Test Bars sintered at 1520°C (2,768°F).
- 3) H.L. Test Bars sintered at 1480°C (2,696°F).
- 4) H.L. Test Bars sintered at 1520°C (2,768°F).

All samples were processed according to the following **furnacing schedule** with sintering temperatures of 1480°C (2,696°F) and 1520°C (2,768°F):

<u>Stage</u>	<u>Beginning Temperature</u>	<u>Ending Temperature</u>	<u>Ramp Rate Per Minute</u>	<u>Atmosphere</u>
Debinding (1)	Room Temp.	400°C (752°F)	2.2°C (4°F)	Dry H ₂
" (2)	400°C (752°F)	600°C (1112°F)	.33°C (.6°F)	Dry H ₂
" (3)	600°C (1112°F)	Room Temp.	5°C (9°F)	Dry H ₂
Presinter 1 (1)	Room Temp.	1200°C (2192°F)	10°C (18°F)	Dry H ₂
" (2)	1200°C (2192°F)	1200°C (2192°F)	Constant - 3 hrs.	Dry H ₂
" (3)	1200°C (2192°F)	Room Temp.	Furnace Cool	Dry H ₂
Presinter 2 (1)	Room Temp.	1400°C (2552°F)	10°C (18°F)	Dry H ₂
" (2)	1400°C (2552°F)	1400°C (2552°F)	Constant - 3 hrs.	Dry H ₂
" (3)	1400°C (2552°F)	Room Temp.	Furnace Cool	Dry H ₂
Sintering	Room Temp.	Sintering Temps.	50°C (90°F)	Wet H ₂
	Sintering Temps.	Sintering Temps.	Constant- 45 min.	Wet H ₂
	Sintering Temps.	Room Temp.	30°C (54°F)	Wet H ₂
Heat Treatment	Room Temp.	1200°C (2192°F)	25°C (45°F)	Vacuum
	1200°C (2192°F)	1200°C (2192°F)	Constant- 3 hrs.	Vacuum
	1200°C (2192°F)	Room Temp.	Furnace Cool	Vacuum

Liquid phase sintering was completed in a wet hydrogen atmosphere to **prevent insitu water vapor** formation. The samples were heated and held three hours in a vacuum atmosphere to remove **hydrogen embrittlement** effects. The sintered samples were **lapped and polished** to facilitate hardness and tensile testing; and were returned to Southwest Research Institute and the U.S. Army for testing.

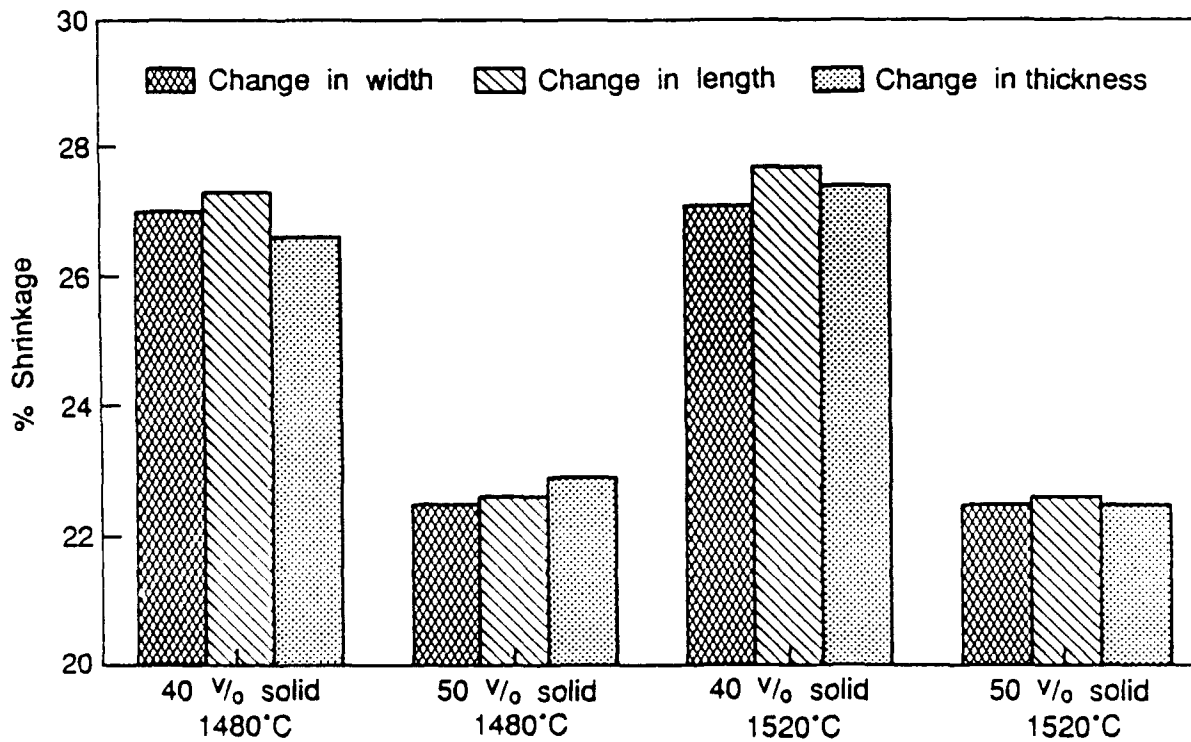
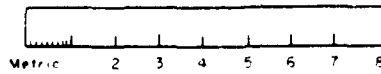


Figure 6. Test Bar Shrinkages.

Part Dimensioning

The dimensions of all samples were measured to determine shrinkage. The shrinkages along the length, width, and thickness of the samples have been plotted in Figure 6. A photograph is also included to illustrate the difference in overall shrinkage for the H.L. (50% powder) and the L.L. (40% powder) samples. It can be observed that the variation in shrinkage between the three directions are within 1%. The actual variation in either width (A), length (B), or thickness (C), for each sample set is also less than 1%.

Material Property Testing

The sintered density of the samples was measured using the water immersion technique with results summarized in Figure 7. All samples had a sintered density greater than 99.6% of theoretical. It is concluded that the PIM of heavy alloys resulted in near fully dense materials with negligible retained porosity.

The hardness of each sample was measured on the flat grip section to preserve the gauge section. At least twelve hardness indentations were taken, and the averages varied between 49.9 - 50.3 HRA. Microhardness measurements with a diamond indent were made on the tungsten grains and the matrix phase in the Vickers scale. The applied load for measuring the microhardness of the tungsten grains was 100 gm. and 10 gm. for the softer matrix phase. An average of four indentations was used to determine the Diamond Pyramid Hardness of the tungsten grains, while only two were used for the matrix phase. The microhardness averages are displayed in Figure 8.

The tensile properties of the samples were tested in an Instron with a crosshead speed of 0.05 inch/min. The yield strength (YS), ultimate tensile strength (UTS), percent elongation, and percent reduction in area of all the specimens are summarized in Figure 9. The yield strength and ultimate tensile strength of the material does not show any significant variation between the four sample sets investigated. The elongation values for the four different specimen sets ranged between 17 and 21%, with the L.L. samples showing slightly higher elongations. The higher solid volume fraction feedstock (0.5) was much more viscous and could result in small defects, which in turn would reflect on the elongation properties of the alloy.

The key result of Phase I was to determine if the tensile properties of the alloys processed by PIM displayed properties comparable to a similar alloy processed by press/sinter. The YS obtained in all four sets varied from 91.1 to 92.6 psi (628 to 638 MPa), the UTS 136.7 to 138.6 psi (942 to 955 MPa), and the elongation from 17 to 21%. These results were equal to results reported in the literature for 95W alloys.

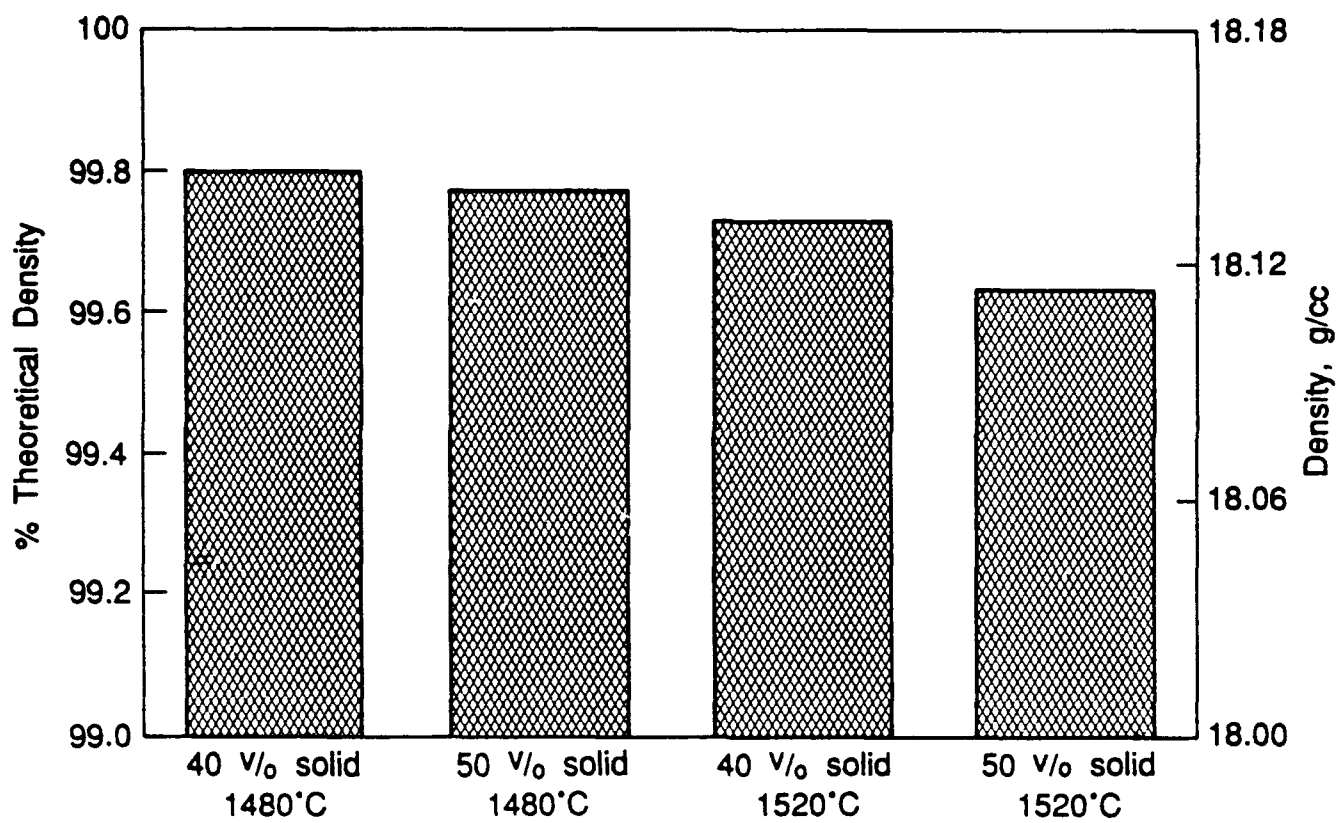


Figure 7. Test Bar Densities.

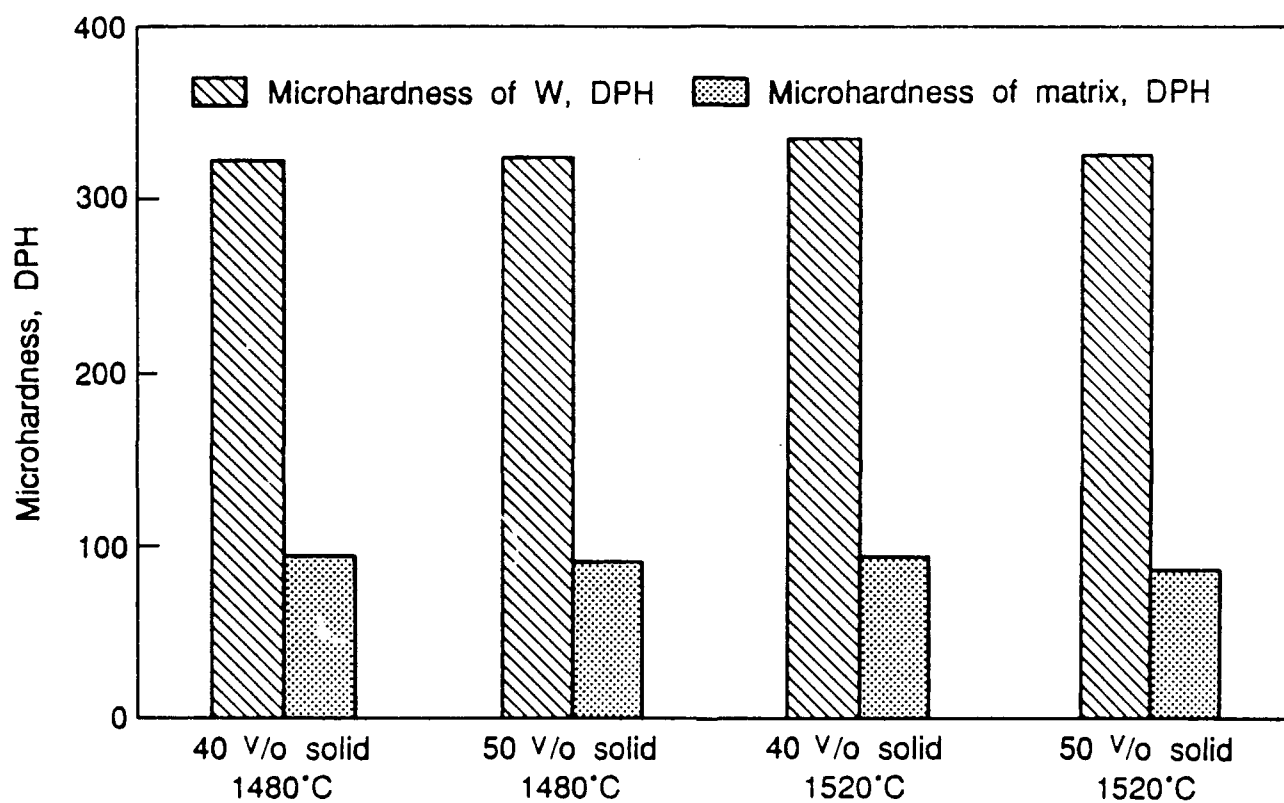


Figure 8. Test Bar Microhardness Data.

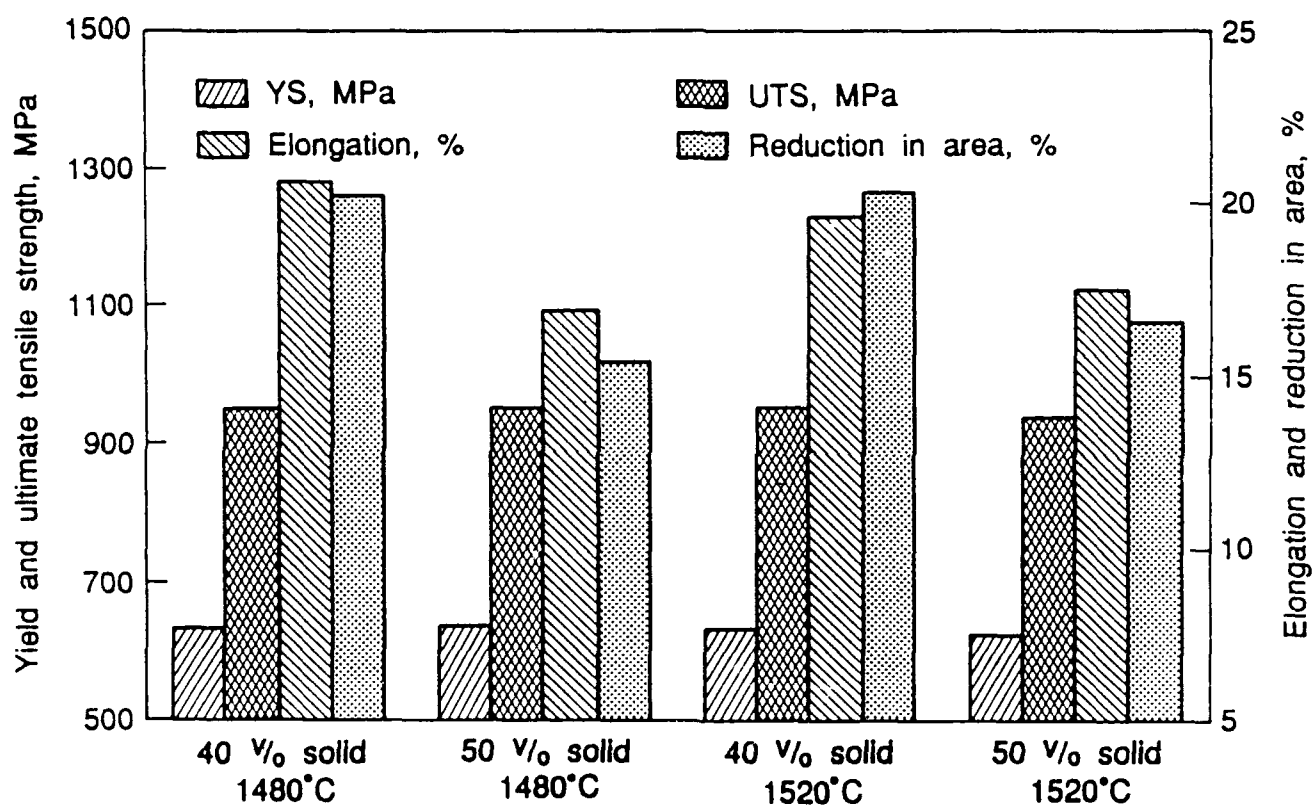
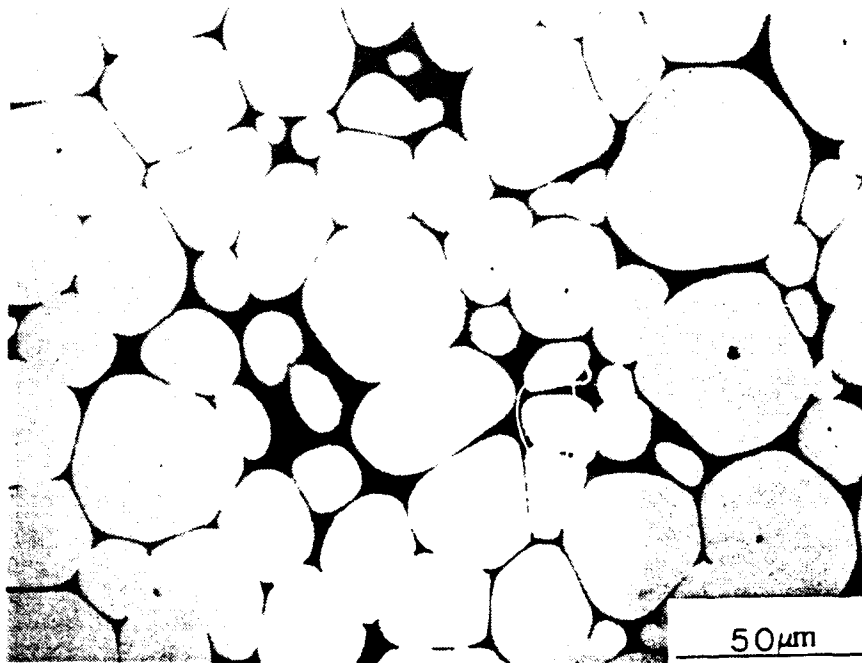


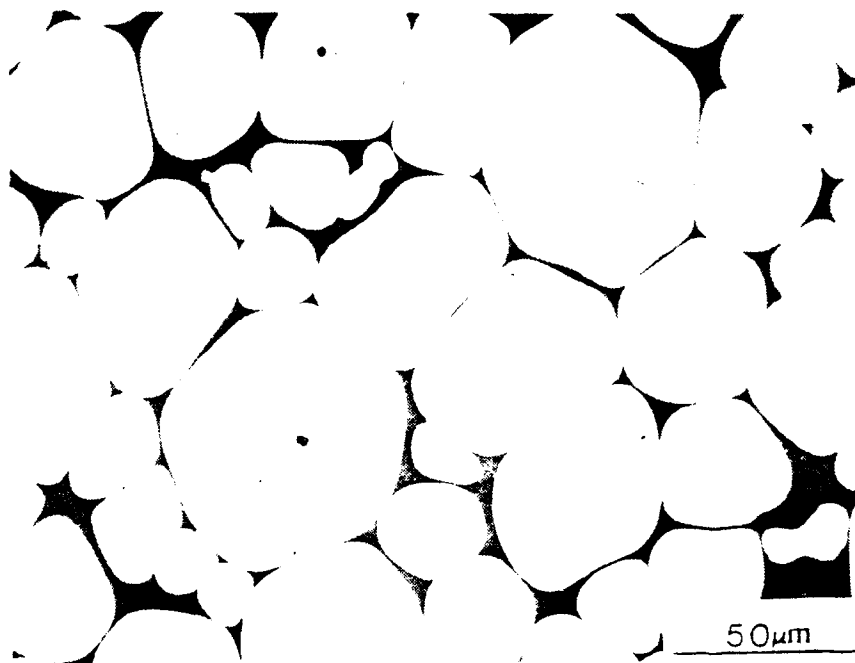
Figure 9. Test Bar Tensile Properties.

A microstructural characterization of the test samples was performed. Small sections removed from one end of a representative sample within each set were mounted, polished, and examined with a Scanning Electron Microscope (SEM). Microstructures were taken from selected areas, and a representative photomicrograph for the sample sets is shown in Figure 10 A and B. The microstructure displays spherical tungsten grains embedded in an alloy matrix of Ni, Fe and W. The microstructures had no porosity, except for the micro-pore that can be seen in the H.L. sample sintered at 1480°C.

The area fraction and the mean intercept length of the tungsten grains were measured from at least three areas with an automated image analyzer. The contiguous tungsten grains were manually dissected before measuring the mean intercept length of the tungsten grains. The results are shown in Figure 11 and are within the normal range of values exhibited by tungsten heavy alloys.

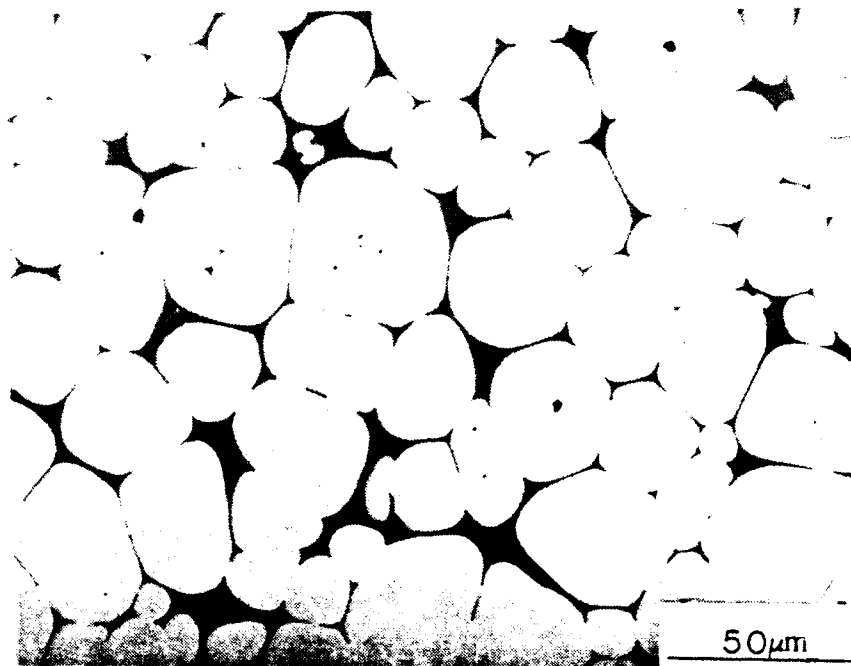


SEM photomicrograph of a heavy alloy sample with 0.4 volume fraction of solid, sintered at 1480°C.

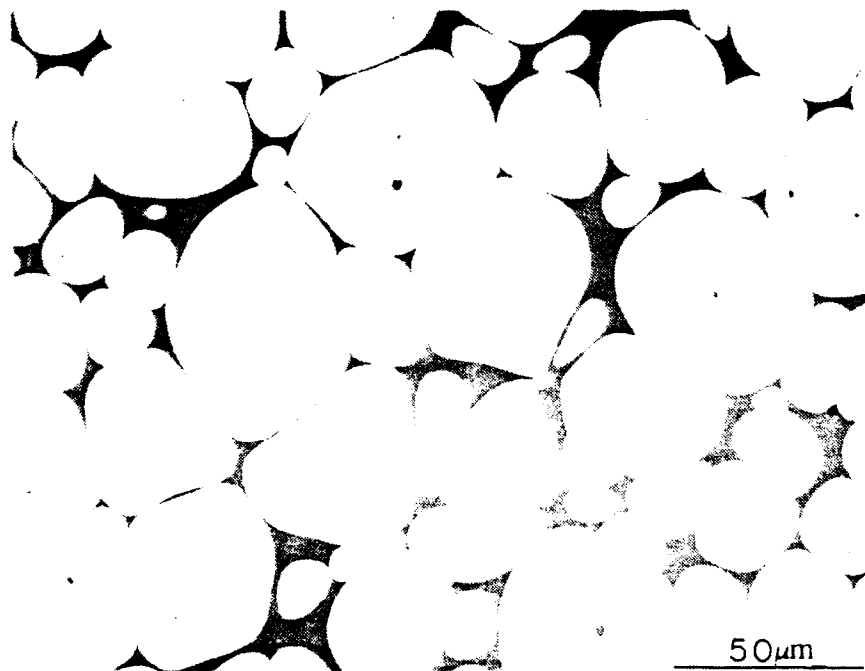


SEM photomicrograph of a heavy alloy sample with 0.4 volume fraction of solid, sintered at 1520°C.

Figure 10A. Microstructure SEM Photomicrographs.



SEM photomicrograph of a heavy alloy sample with 0.5 volume fraction of solid, sintered at 1480°C.



SEM photomicrograph of a heavy alloy sample with 0.5 Volume fraction of solid, sintered at 1520°C.

Figure 10B. Microstructure SEM Photomicrographs.

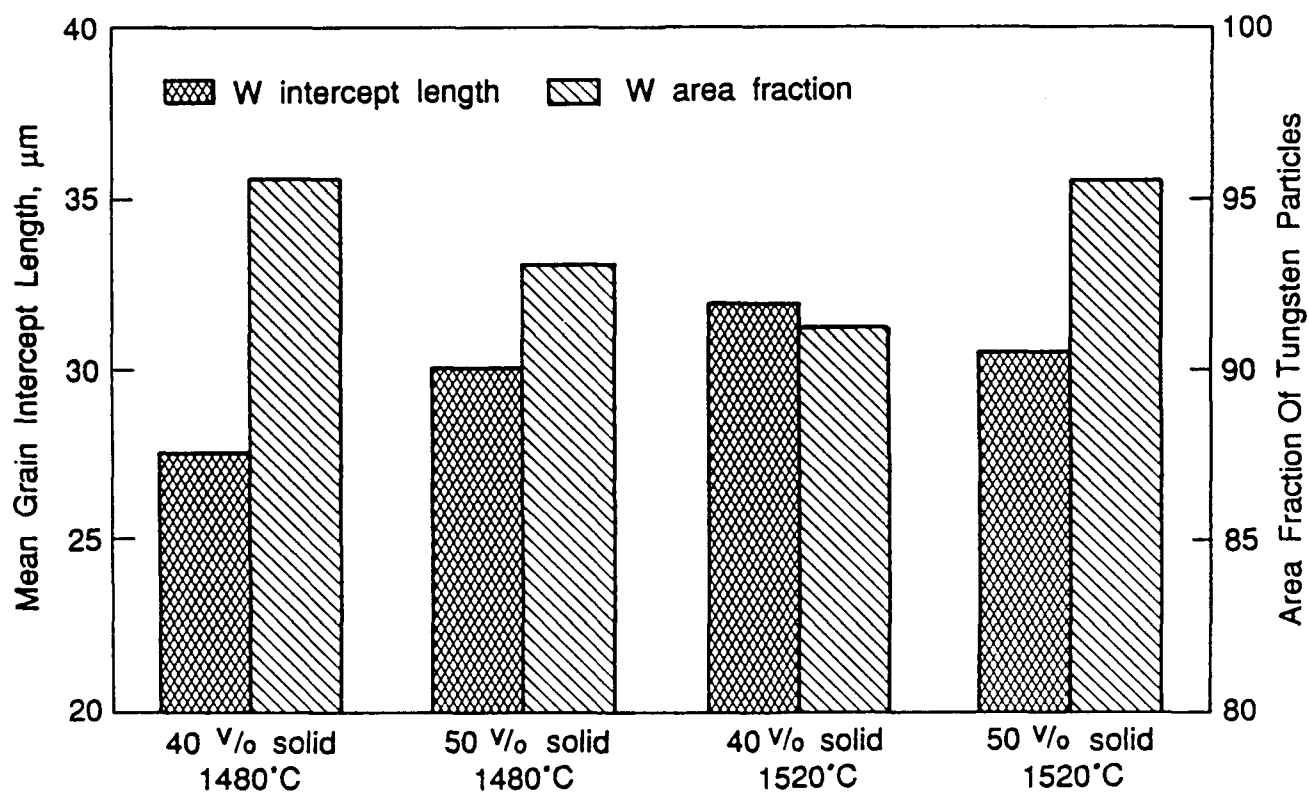


Figure 11. Tungsten Grain Characteristics.

Chemical Analysis

The alloy **chemistry** of the **matrix phase** was evaluated by Energy Dispersive Spectroscopic (EDS) Analysis and is illustrated by the EDS spectra in Figures 12 A and B for the four sample sets.

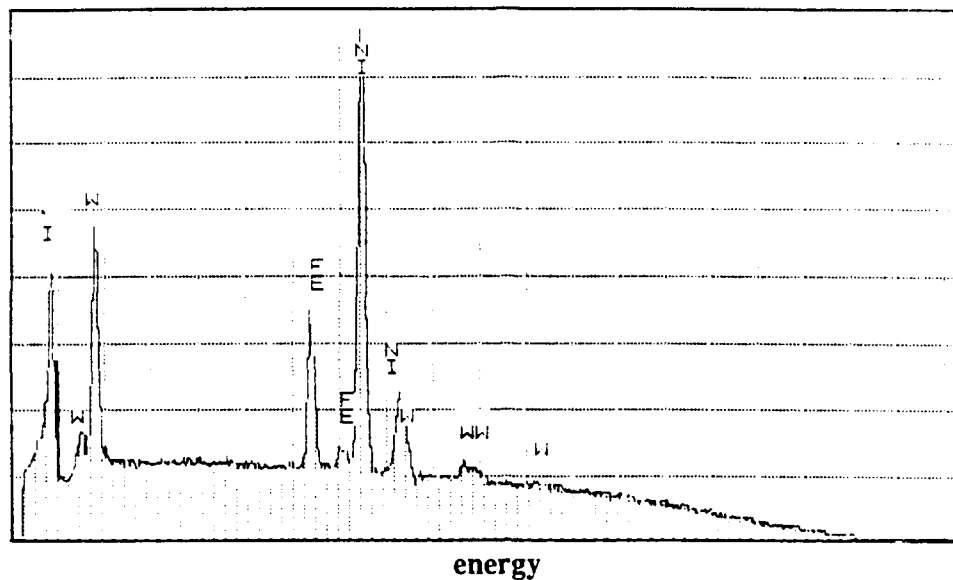
Figure 13 shows the EDS spectra taken on a representative **tungsten grain** which reveals a pure tungsten content. The quantitative analysis of the **matrix chemistry** of all the samples showed the matrix alloy to have a composition of approximately 51Ni-38W-11Fe as illustrated in Figure 14.

Carbon control in PIM of heavy alloys is a major concern since both carbon and oxygen can play a major role in decreasing resultant properties. The sintered PIM material and elemental powder blend were chemically analyzed by J. Dirats and Company (Westfield, MA) for both carbon and oxygen. The results indicated that the PIM processed materials did not result in any carbon or oxygen pick-up per the results presented in Figure 15. The notations included in this figure are summarized as follows:

<u>Notation</u>	<u>Meaning</u>
Sintered Part	Furnace Test Specimen
Powder S/N 1	Powdered Master Alloy - First Random Sample
Powder S/N 2	Powdered Master Alloy - Second Random Sample

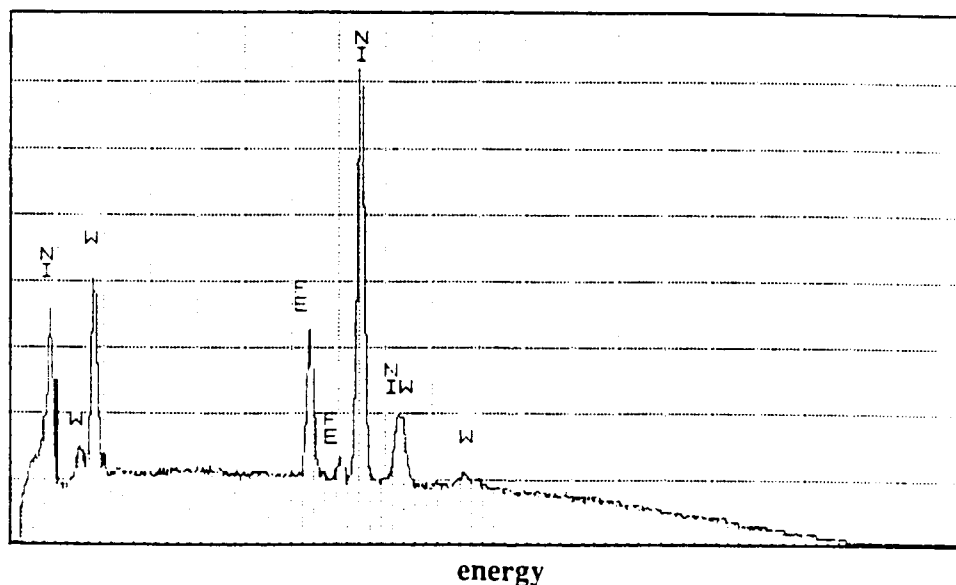
During presintering and sintering, the reducing action of the hydrogen causes significant reduction in carbon and oxygen content due to the reactions that form water vapor, carbon dioxide, and carbon monoxide. This subsequently results in lower oxygen and carbon contents.

Relative
Counts



EDS analysis of the matrix of a heavy alloy with 0.4 volume fraction of solid, sintered at 1480°C.

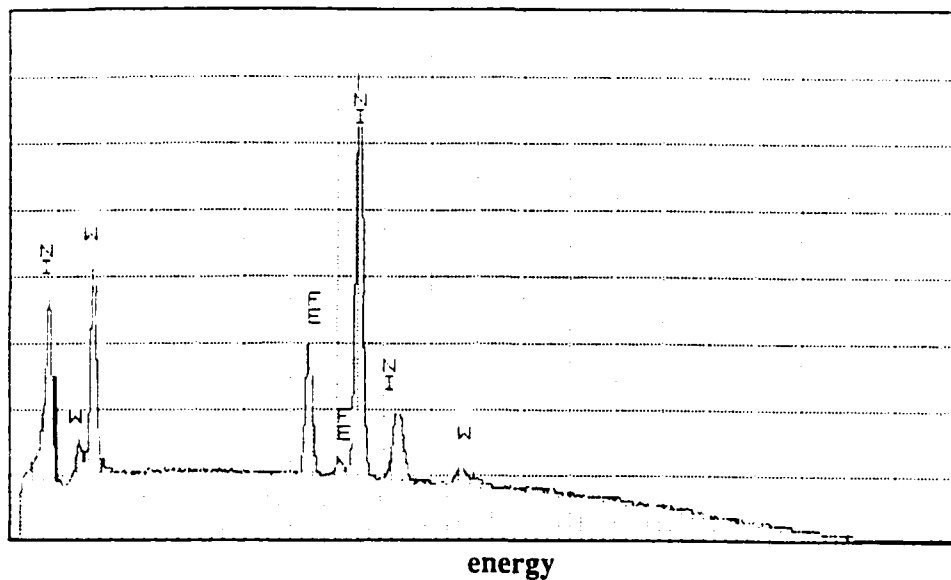
Relative
Counts



EDS analysis of the matrix of a heavy alloy with 0.4 volume fraction of solid, sintered at 1520°C.

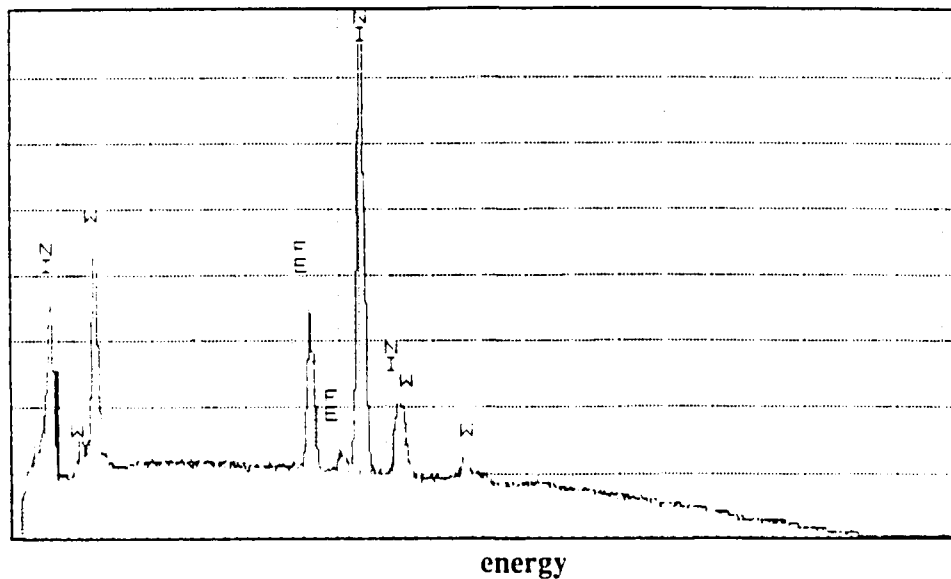
Figure 12A. Matrix Phase Alloy Chemistry.

Relative
Counts



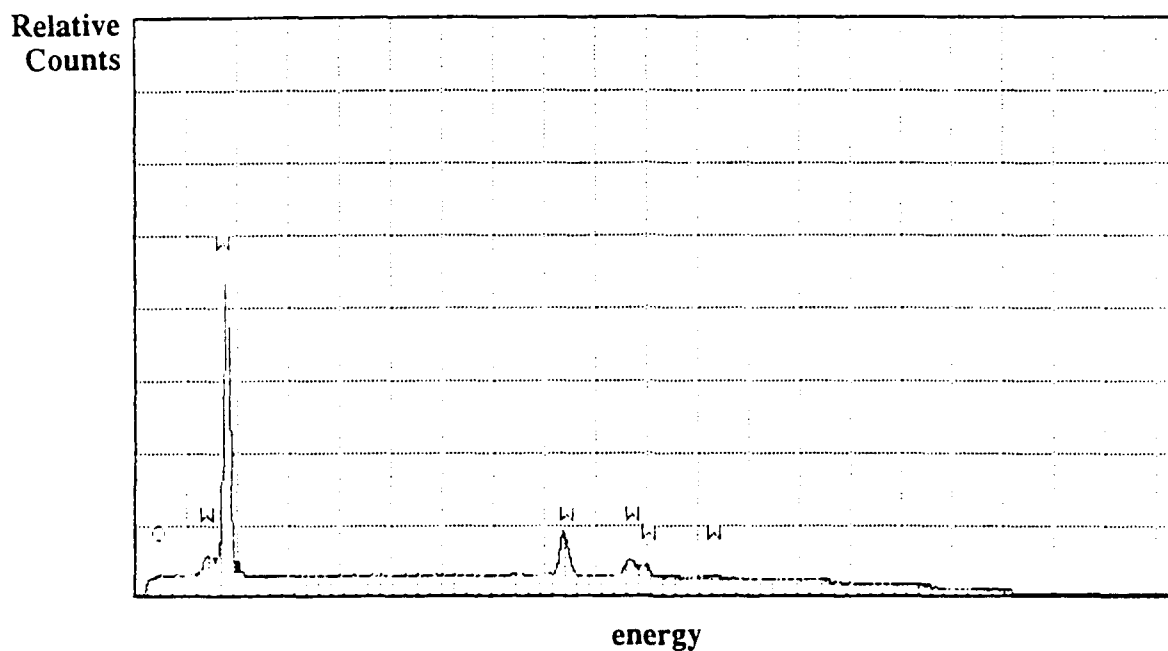
EDS analysis of the matrix of a heavy alloy with 0.5 volume fraction of solid,
sintered at 1480°C.

Relative
Counts



EDS analysis of the matrix of a heavy alloy with 0.5 volume fraction of solid,
sintered at 1520°C.

Figure 12B. Matrix Phase Alloy Chemistry.



EDS analysis of a tungsten grain.

Figure 13. Tungsten Grain Alloy Chemistry.

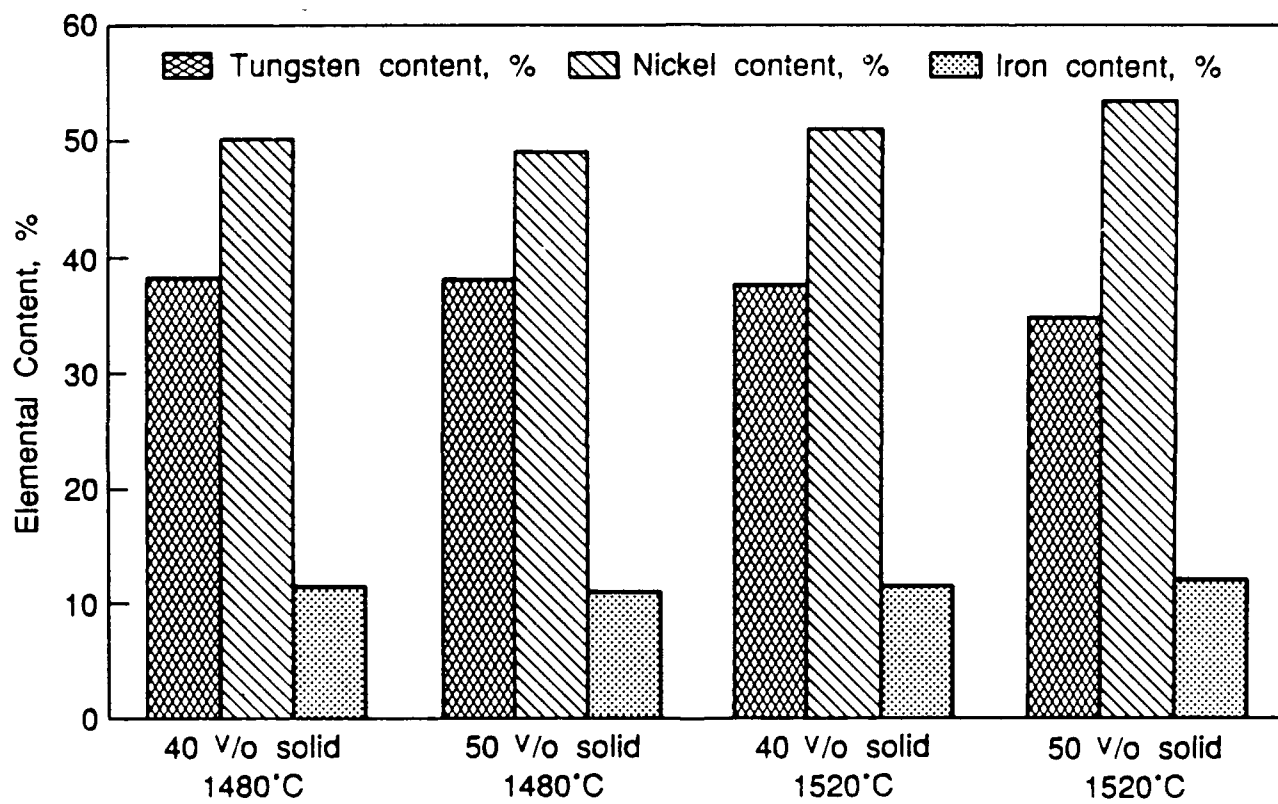


Figure 14. Matrix Chemistry - Quantitative Analysis.

Gary Allen
Technology Associates Corp.
17911 Sampson Lane
Huntington Beach, CA 92647

Report Number 139540
Report Date 7-MAY-91
Client Number **816800
Client Order 2508

RECEIVED 1 Sintered Part and 2 Samples Powder
IDENT AS *
MATERIAL W-Ni-Fe Alloy
CONDITION *
TEST TO *
TEST PER Client Instructions
PURPOSE *

PHONE 714-842-8882

PROPERTIES AS SUPPLIED

QUANTITATIVE ANALYSIS BY ICP, COMB.

Sample	SINTERED PART	POWDER S/N 1	POWDER S/N 2
%			
W	95.19	94.66	94.71
Ni	3.75	4.01	3.98
Fe	0.99	1.03	1.03
N	0.0050	0.0301	0.0218
O	0.0073	0.2529	0.2465
Others:			
Ti	0.02	<0.01	<0.01
Ta	0.02	<0.01	<0.01
Co	0.02	<0.01	<0.01
C	<0.01	0.01	0.01
S	0.002	0.004	0.005

For Info

For Info

For Info

The symbol < signifies not detected at the detectability limit indicated.

WE CERTIFY THIS IS A TRUE COPY OF OUR RECORDS

Signed for J. Dirats and Co. by Eric Dirats, Audit Manager

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Figure 15. Chemical Analysis.

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HEAVY ALLOYS: SBIR PHASE I
Gary M. Allen
Technology Associates Corporation
17911 Sampson Lane
Huntington Beach, CA 92647
Technical Report TR 91-37, October 1991, 34 pp -
illus., Contract DAAL04-90-C-0018
Final Report - 8/20/90 - 6/20/91

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